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# Correlations Among Trihalomethane (THM) and Total Organic Halide (TOX) Formation in Water Treatment Facilities

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16. ABSTRACT
In determining the extent to which correlations exist among certain parameters (TOX, TTHM, TOC, UV absorbance, THM formation potential, and TOX formation potential), samples of raw water and treated water, including samples from the distribution systems, were collected from 12 full-scale systems in six states. The ratio of TTHM/TOX in surface waters treated by conventional coagulation, sedimentation, and filtration, at near neutral pH values (approximately 3:1), corresponds closely with the THMFP/TOXFP ratio in raw waters after a chlorine contact of 7 days at pH 7. Hence, the formation potentials measured at pH 7 provide a good indication of the TTHM/TOX distribution to be expected in waters treated at near neutral pH.

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EPA/600/D-86/203

# CORRELATIONS AMONG TRIHALOMETHANE (THM) AND TOTAL ORGANIC HALIDE (TOX) FORMATION IN WATER TREATMENT FACILITIES

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Several researchers have shown that when raw water or humic substances extracted from natural waters are chlorinated, other halogenated organic compounds in addition to the trihalomethanes are produced. The nature of the specific organic halides formed, the possibility of any adverse health effects associated with their presence, and the extent of their formation relative to the regulated THMs is an active area of current research.

TOX, representing the concentration of total organic halides, is a collective parameter which is finding increased use as a surrogate for potentially harmful halogenated organic substances in drinking water. (1) TOX/THM ratios of 3 to 10 have been measured for various waters, depending upon treatment conditions. For example, Reckhow and Singer (2) showed that TOX to $CHCl_3$ ratios ranged from 3 to 5 when 4.1 mg/l (as C) of an aqueous fulvic acid extract was treated with excess chlorine at pH 7.0. The ratio of TOX to $CHCl_3$ varied, depending upon the chlorine contact time, pH, and $Cl_2$/C ratio. Similar observations were reported by Miller and Uden. (3) Reckhow and Singer (2) also showed that TOX formation from the chlorination of a variety of aquatic humic and fulvic extracts from several different sources was relatively uniform, with approximately 220 ug TOX produced per mg TOC due to chlorination at pH 7.0, at a $Cl_2$/C ratio of about 4 or 5 to 1 (mg/mg basis), and a 72-hour contact time. (See Table 1). They also showed that, regardless of the source, $CHCl_3$ comprised approximately 20% of the TOX, trichloroacetic acid accounted for about 18%, dichloroacetic acid contributed approximately 6%, with trichloroacetone and dichloroacetonitrile comprising an average of less than 1%, as illustrated in Table 1. Altogether, these species accounted for 44% of the measured TOX. These same compounds have been found in a variety of finished drinking waters we have examined. (See Table 2.) (4)

While other organic halides have been identified in chlorinated humic and fulvic solutions and in treated drinking water (5,6), their concentrations have not been adequately quantified, although they are believed to be present at very low concentrations. In all such studies, no more than about 50% of the TOX content has been accounted for, despite evidence that several of these halogenated by-products of water chlorination may be harmful to man.



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Accordingly, since the concentration of TOX is readily measurable, (1,7) this surrogate parameter has become the focus of increased attention. While TOX itself is currently not regulated in finished drinking water, good practice suggests that in controlling THM formation, TOX formation should be controlled as well. Note that TOX formation tends to parallel THM formation for the several utilities depicted in Table 2.

The objective of this paper is to determine the extent to which correlations exist among the following parameters: TOX, TTHM, TOC, UV absorbance, THM formation potential and TOX formation potential. The study is based upon actual full-scale treatment plant data collected from twelve utilities in six states (Indiana, Delaware, Virginia, North Carolina, South Carolina, and Florida). Several of these utilities have modified their treatment operations during the course of this study by moving the point of chlorine addition and pre-treating with $KMnO_4$, $ClO_2$ or $O_3$, or by adding $NH_3$ to the treated water. Samples of raw water as well as treated water were collected, including samples from the distribution systems. Data were collected over all four seasons, from December 1983 through March, 1985.

## PROCEDURES

Three sets of samples were collected at each of the facilities visited. One set was acidified with phosphoric acid (1 ml of 1 M $H3PO_4$ per 40 ml sample) at the time of collection, for subsequent measurement of TOC and UV absorbance. A second set of samples was collected in 40 ml Pierce bottles with screw-on caps containing a Teflon liner. These were filled headspace-free, dosed with crystals of sodium sulfite, and sealed for subsequent analysis of TTHM and TOX. These represent Instantaneous THM and TOX measurements. The third set of samples was also collected in Pierce bottles, but these samples were buffered at pH 7 with a phosphate buffer and dosed with excess NaOCl at the time of collection (approximately 2-3 mg $Cl_2$ per mg TOC). The bottles were sealed headspace-free and stored for 7 days at room temperature, after which they were analyzed for TTHM and TOX. These represent Terminal THM and TOX measurements. THM and TOX Formation Potentials were calculated as the differences between the respective Terminal and Instantaneous concentrations. These definitions are consistent with earlier ones used by this research group. (8)

TTHMs were analyzed by the liquid-liquid extraction procedure. (9) TOC was measured using a Beckman 915B Carbon Analyzer. For waters with high alkalinity, inorganic carbon was purged by bubbling with purified nitrogen and then measuring total carbon. For samples with low alkalinity, inorganic carbon was measured and subtracted from the total carbon measurements. UV absorbance was measured at a wavelength of 254 nm in a 1 cm cell using a Perkin-Elmer Lambda 3 UV-Visible Spectrophotometer; raw water samples containing appreciable turbidity were filtered first through Gelman A/E glass-fiber filters which had been pre-rinsed with organic-free water. TOX was measured using a Dohrmann DX-20 Total Organic Halide Analyzer system. (7) All THM and TOC analyses were performed in duplicate; replicate analyses for TOX and UV absorbance were performed only as needed.

Table 3 illustrates the analytical program for each sampling visit. Terminal measurements were made for only raw and settled water in order to determine the extent of removal of precursors by coagulation and settling. TTHM and TOX analyses were performed on both the raw and treated water in order to determine the progression of organic halide formation through the treatment plant and on out into the distribution system.

Table 4 shows data collected from one of our sampling trips. This utility draws water from a river with a fairly high color and TOC concentration; the raw water is coagulated with alum and chlorine is applied after sedimentation. It is obvious from the table that chloroform is the principal trihalomethane species produced, that the formation of TOX tends to parallel THM formation, and that the removal of THM and TOX precursors by coagulation and settling, as represented by the respective raw and settled water terminal values, are about the same and are similar to the removal of TOC.

Accordingly, the relationships tested in this paper are shown in Table 5. Linear regression analysis was performed using a standard statistical analysis package. (10) The latter three sets of correlations were attempted in order to determine if the average TTHM and TOX concentrations in the distribution system could be predicted knowing the TOC concentration or the respective Terminal values of the raw water.

## RESULTS AND DISCUSSION

Figure 1 shows the relationship between the trihalomethane formation potential (ug/l TTHM) and the total organic halide formation potential (ug/l as $Cl_2$) of the raw waters sampled during this study. In all, 42 pieces of data are represented. The slope of the line of best fit is 0.345 +/- 0.015. In all cases reported, $CHCl_3$ comprises in excess of 90% of the TTHM. The correlation coefficient R has a value of 0.965, demonstrating the goodness of fit. The intercept is relatively small; if the best fit line is forced through the origin, the slope is essentially unchanged (0.342 +/- 0.008). Converting the THMFP to an equivalent concentration as $Cl_2$, the THMFP represents 30.7% of the TOXFP. If the analysis is expanded to include data for both raw water and settled water, the line of best fit drawn through the origin is not significantly different (slope is 0.349 +/- 0.007).

The correlation between the TOX formation potential and TOC of the raw water is illustrated in Figure 2. The correlation coefficient is lower than for Figure 1 but is still significant. Note the increased divergence from the

line as the magnitude of the TOC increases. The slope of the line is 148 +/- 17.5. If the line is forced through the origin, the slope becomes 159 +/- 9.1. This indicates that a water containing 10 mg/l of TOC has a 7-day TOX formation potential of 1590 ug/l at pH 7. If data for both raw and settled water are included, the line through the origin has a slope of 160 +/- 6.6.

Singer et al. (8) and Edzwald et al. (11) have shown good correlations between the THMFP and TOC of raw drinking waters. The results of a parallel analysis of data for THMFP and TOC for raw and settled water samples collected in this study yielded the following correlation:

THMFP (ug/l) = 53.4 +/- 4.3 TOC (mg/l) + 45.5  R = 0.823

This relationship is consistent with the earlier ones reported. (8,11)

Figure 3 demonstrates the correlation between the TOX formation potential and the UV absorbance (at 254 nm, 1 cm light path) of the raw water. The strong correlation between the two (R = 0.889) agrees with the strong correlation between the THMFP and UV absorbance of raw drinking water reported by Singer et al. (8) and Edzwald et al. (11) In this study, the correlation between THMFP and UV absorbance for both raw and settled water was:

THMFP (ug/l) = (2218 +/- 254) UV + 94  R = 0.872

It should be noted that in all of the above cases, the lines forced through the origin are not significantly different from the lines determined by least squares analysis.

While the relationships above all pertain to formation potentials and their utility as surrogates for the precursor content of a water, a more practical concern is the relationship between the instantaneous TTHM and TOX concentrations in the treated water, including water in the distribution system. Figure 4 shows the strong correlation between TTHM and TOX in the treated water. If all 198 data points are considered, the slope of the line is 0.431 +/- 0.015 and the correlation coefficient is 0.903. If the line is forced through the origin, the slope is not significantly different.

It should be noted that the data represented in Figure 4 are for all twelve of the treatment facilities sampled. It is well known, based upon chlorination studies of humic material in model systems (2,3,12,13) that high pH tends to favor $CHCl_3$ formation over the formation of trichloroacetic acid and other organic halides. Accordingly, water plants practicing precipitative softening at pH values in excess of 9.5-10 are likely to have a higher fraction of TOX attributable to trihalomethanes than plants treating surface waters by conventional coagulation, sedimentation, and filtration, where pH tends to range from about 5.5 to 7.5 during the course of treatment and distribution.



Examination of the data represented in Figure 4 indicates that most of the points on the upper portion of the line are derived from samples collected from precipitative softening facilities in Florida. These data have a strong influence over the least squares line represented by the relationship shown in Figure 4. If the Florida entries are removed from the data set, Figure 5 shows that the regression line is shifted downward, and the new relationship has a slope of 0.323 +/- 0.015, with a correlation coefficient of 0.857. If the line is forced through the origin, again the slope is not significantly different.

If the TTHM concentrations in Figure 5 are converted to an equivalent chlorine basis, these results indicate that the THMs comprise approximately 29% of the TOX in surface waters treated by conventional coagulation, sedimentation, and filtration. These results are remarkably consistent with those shown in Figure 1 for the THM and TOX formation potentials, demonstrating that formation potentials measured at pH 7 provide a good indication of the THM/TOX distribution to be expected in waters treated at near neutral pH. The results also suggest that, since the THMs are the principal volatile organic halides in treated drinking water, the non-volatile organic halides are present at concentrations approximately 2.5 times the TTHM concentration, on an equivalent chlorine basis.

For the Florida utilities, where treatment at pH values well in excess of 9.5 is practiced, the TTHM comprise approximately 45% of the TOX (39% on a chlorine equivalent basis), as shown in Figure 6.

Attempts to correlate the average TOX concentration in the distribution system with the TOC or Terminal TOX concentration in the raw water were not very successful, as shown in Figures 7 and 8, respectively. Similar attempts to correlate the average TTHM concentration in the distribution system with raw water TOC or Terminal TTHM were also unsuccessful (correlation coefficients were 0.445 and 0.206, respectively). This is not surprising in view of the different treatment conditions at each of the plants which influences reaction kinetics, e.g. pH, temperature, chlorine contact time, etc. Even if the Florida data are deleted from the data set, the correlation is only slightly improved (see Figure 9).

The lack of a good correlation suggests that it is not possible to predict the TOX and TTHM concentrations in the water distribution system simply by knowing the formation potentials or TOC concentration in the raw water. However, it may be possible, by using multiple regression analysis, that an acceptable predictive equation can be developed relating the TOX and TTHM concentrations in the distribution system to some function involving the residence time in various of the treatment units and the distribution system, the treatment and distribution system pH, temperature, chlorine dose, and TOC concentration. Such an attempt was made for TTHM concentrations in

finished water with some success by Barry (14) and Edzwald et al. (11) The data collected in this study are currently being analyzed using such multiple regression techniques.

Finally, Table 6 illustrates the average percent removals of TOC, TOXFP, and THMFP by coagulation and settling for all the utilities included in this study. The percent removals averaged approximately 50% for each of the parameters. This is consistent with earlier observations summarized by Singer (15) and attests to the importance of moving the point of chlorine application in the control of TOX as well as TTHM. It should be noted that the removals of TOC and TOX and THM precursors were lower for the Florida utilities where removal was accomplished at the alkaline pHs of precipitative softening. Insufficient data are available to calculate a meaningful percent removal for the Florida utilities.

## CONCLUSIONS

For the utilities monitored in this study, the following conclusions can be reached:

1. For surface waters treated by conventional coagulation, settling and filtration, at near neutral pHs, the TOX/TTHM ratio in the treated water, including the distribution system, is approximately 3:1. On a chlorine equivalent basis, the TTHMs comprise approximately 29% of the total organic halide concentration. The results suggest that the concentration of non-volatile organic halides, such as trichloro- and dichloroacetic acid and many others which have not been identified as yet, are approximately 2.5 times the concentration of TTHMs.

2. In waters subject to precipitative softening at alkaline pHs, the TTHMs on a chlorine equivalent basis comprise approximately 39% of the total organic halide concentration.

3. The ratio of TTHM/TOX in surface waters treated by conventional coagulation, sedimentation, and filtration at near neutral pH values corresponds closely to the THMFP/TOXFP ratio in raw waters after a chlorine contact time of seven days at pH 7. Hence, the formation potentials measured at pH 7 provide a good indication of the TTHM/TOX distribution to be expected in waters treated at near neutral pH.

4. The instantaneous TOX and TTHM concentrations in the distribution system do not correlate well with the TOC, TOXFP or THMFP of the raw water.

5. Approximately 50-55% of the TOC, TOXFP, and THMFP of raw drinking water can be removed by conventional coagulation and sedimentation.



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TABLE 1

FORMATION OF ORGANIC HALIDES FROM AQUATIC HUMIC EXTRACTS (AFTER RECKHOW AND SINGER.[2])

| SOURCE | TOX ug/mg C | PERCENT OF TOX | | | | |
|---|---|---|---|---|---|---|
| | | $CHCl_3$ | TCAA | DCAA | TCAC | DCAN |
| BLACK LAKE | | | | | | |
| FULVIC ACID | 208 | 20.8 | 15.7 | 4.7 | 0.47 | 0.20 |
| HUMIC ACID | 288 | 21.1 | 22.3 | 5.7 | 0.42 | 0.34 |
| COAL CREEK | | | | | | |
| FULVIC ACID | 232 | 19.5 | 18.7 | 5.9 | 0.39 | 0.21 |
| HUMIC ACID | 268 | 19.9 | 22.6 | 5.7 | 0.24 | 0.25 |
| OGEECHEE RIVER | | | | | | |
| FULVIC ACID | 216 | 20.6 | 18.3 | 5.6 | 0.50 | 0.18 |
| HUMIC ACID | 262 | 20.2 | 22.5 | 6.7 | 0.21 | 0.22 |
| OHIO RIVER | | | | | | |
| FULVIC ACID | 161 | 18.0 | 12.3 | 5.7 | 0.63 | 0.27 |
| HUMIC ACID | 232 | 18.1 | 19.8 | 6.4 | 0.37 | 0.45 |
| MISSOURI RIVER | | | | | | |
| FULVIC ACID | 136 | 20.2 | 11.3 | 4.8 | 0.82 | 0.28 |
| HUMIC ACID | 230 | 19.0 | 17.8 | 5.8 | 0.36 | 0.63 |
| AVERAGE | 223 | 19.7 | 18.1 | 5.7 | 0.44 | 0.30 |

[a] 20 mg/l HOCl, pH 7, 72 hrs., 20°C

TABLE 2

OCCURRENCE OF ORGANIC HALIDES IN SELECTED WATER TREATMENT SYSTEMS (AFTER RECKHOW.[4])

| UTILITY | DATE | TOX ug/l | $CHCl_3$ ug/l | TTHM ug/l | TCAA ug/l | DCAA ug/l | TCAC ug/l | DCAN ug/l |
|---|---|---|---|---|---|---|---|---|
| #1 | 2/23/83 | 152 | 32 | 39 | 15 | 8 | 1.8 | 3.2 |
| | 1/27/84 | 155 | 34 | 40 | 18 | 31 | 3.1 | 2.9 |
| #2 | 2/23/83 | 298 | 56 | 66 | 26 | 12 | 3.1 | 5.1 |
| #3 | 1/18/84 | 1234 | 295 | 377 | 103 | 78 | >1.8 | >6.5 |
| #4 | 1/6/84 | 549 | 91 | 104 | 32 | 42 | >0.9 | >2.0 |
| #5 | 1/17/84 | 319 | 84 | 90 | 64 | 52 | - | - |
| #6 | 1/18/84 | 460 | 79 | 85 | 53 | 79 | - | - |

[a] SAMPLES COLLECTED FROM WATER DISTRIBUTION SYSTEMS.

TABLE 3

SAMPLING PROGRAM

| | TOC | UV | TTHM | TERM THM | TOX | TERM TOX |
|---|---|---|---|---|---|---|
| RAW WATER | X | X | X | X | X | X |
| SETTLED WATER | X | X | X | X | X | X |
| FILTERED WATER | X | X | X | | X | |
| FINISHED (TAP) WATER | | | X | | X | |
| DISTRIBUTION SYSTEM | | | X | | X | |

TABLE 4

ILLUSTRATIVE DATA COLLECTED DURING SAMPLING TRIP

| SAMPLE | FREE Cl2 mg/l | TOC mg/l | UV-254 | CHCl3 ug/l | TTHM ug/l | TOX ug/l | TERM THM ug/l | TERM TOX ug/l |
|---|---|---|---|---|---|---|---|---|
| RAW WATER | - | 8.7 | 0.277 | 0 | 0 | 30 | 725 | 2050 |
| SETTLED WATER | - | 4.3 | 0.078 | 2 | 4 | 57 | 343 | 1024 |
| FILTERED WATER | 0.2 | 4.3 | 0.050 | 52 | 60 | 206 | - | - |
| FINISHED WATER | 1.8 | - | - | 120 | 133 | 397 | - | - |
| DS1 | 1.3 | - | - | 120 | 134 | 433 | - | - |
| DS2 | 0.2 | - | - | 171 | 191 | 434 | - | - |



TABLE 5

RELATIONSHIPS TESTED

| | | | |
|---|---|---|---|
| THMFP | vs | TOXFP | RAW WATER |
| THMFP, TOXFP | vs | TOC | RAW WATER |
| THMFP, TOXFP | vs | UV | RAW WATER |
| TTHM | vs | TOX | TREATED WATER (SET, FILT, FIN, DS) |
| TTHM(DS), TOX(DS) | vs | TOX (RAW) | |
| TTHM(DS) | vs | TERM THM (RAW) | |
| TOX(DS) | vs | TERM TOX (RAW) | |

TABLE 6

REMOVAL OF TOC, TOXFP, AND THMFP BY COAGULATION AND SETTLING

| TOC % | TOXFP % | THMFP % | |
|---|---|---|---|
| 53.2 ± 18.2 n = 41 | 51.8 ± 17.9 n = 37 | 49.8 ± 15.1 n = 37 | All Utilities |
| 55.1 ± 16.7 n = 33 | 55.0 ± 17.1 n = 29 | 53.1 ± 13.0 n = 29 | All Utilities exc. FLA. |

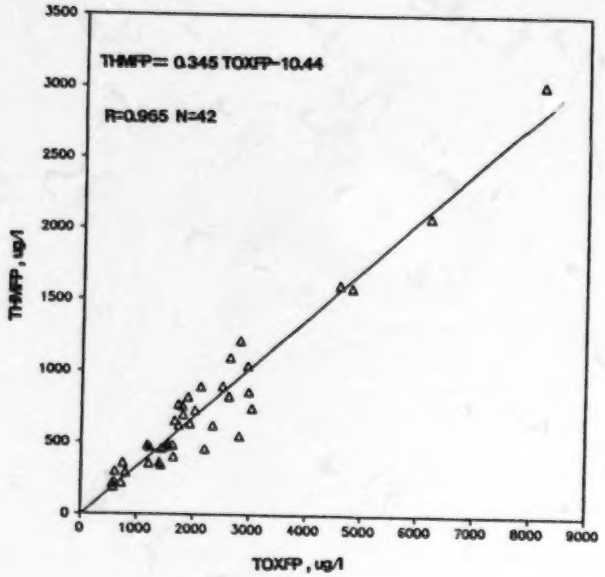


FIGURE 1. RELATIONSHIP BETWEEN TRIHALOMETHANE FORMATION POTENTIAL AND TOTAL ORGANIC HALIDE FORMATION POTENTIAL FOR RAW WATERS

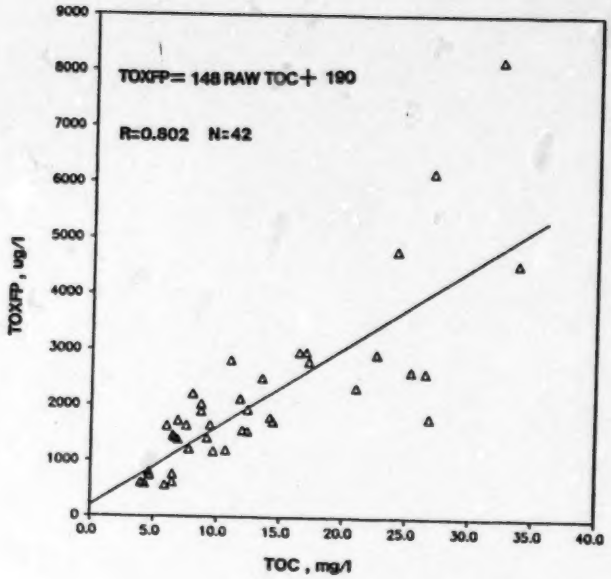


FIGURE 2. RELATIONSHIP BETWEEN TOTAL ORGANIC HALIDE FORMATION POTENTIAL AND TOTAL ORGANIC CARBON CONCENTRATION FOR RAW WATERS

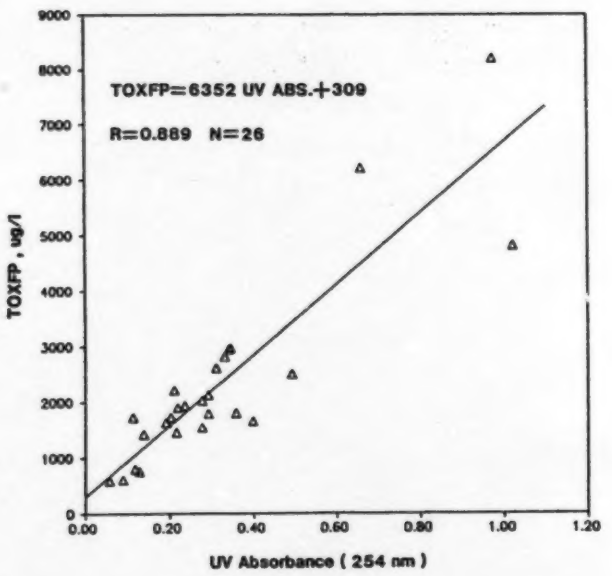


FIGURE 3. RELATIONSHIP BETWEEN TOTAL ORGANIC HALIDE FORMATION POTENTIAL AND ULTRAVIOLET ABSORBANCE FOR RAW WATERS

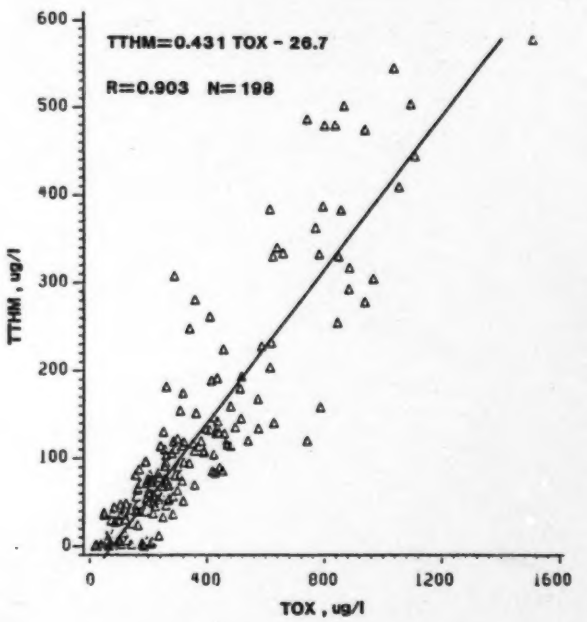


FIGURE 4 RELATIONSHIP BETWEEN INSTANTANEOUS TOTAL TRIHALOMETHANE CONCENTRATION AND TOTAL ORGANIC HALIDE CONCENTRATION IN TREATED WATER

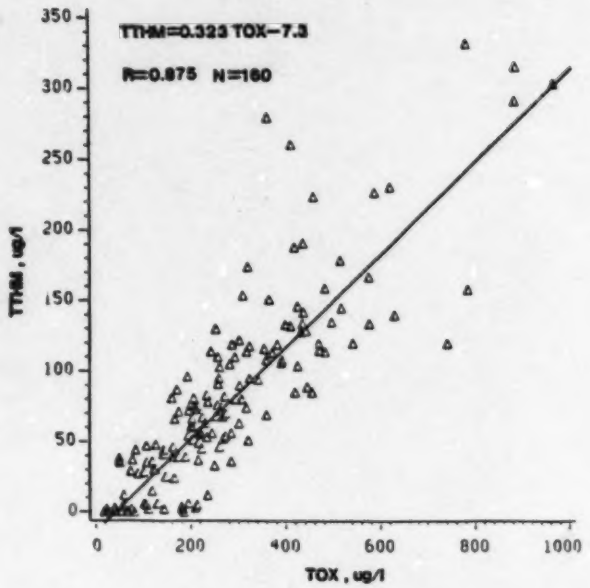


FIGURE 5. RELATIONSHIP BETWEEN TOTAL TRIHALOMETHANE CONCENTRATION AND TOTAL ORGANIC HALIDE CONCENTRATION IN SURFACE WATERS TREATED BY CONVENTIONAL COAGULATION SETTLING AND FILTRATION

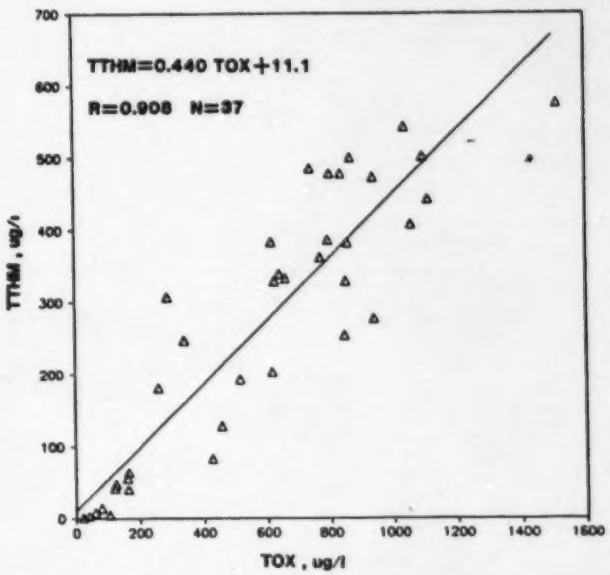


FIGURE 6. RELATIONSHIP BETWEEN TOTAL TRIHALOMETHANE CONCENTRATION AND TOTAL ORGANIC HALIDE CONCENTRATION IN WATERS TREATED BY PRECIPITATIVE SOFTENING

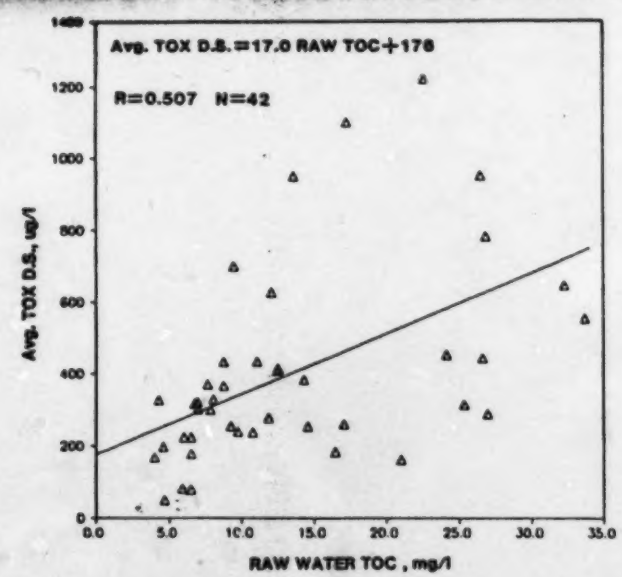


FIGURE 7. RELATIONSHIP BETWEEN INSTANTANEOUS TOTAL ORGANIC HALIDE CONCENTRATION IN DISTRIBUTION SYSTEM AND TOTAL ORGANIC CARBON CONCENTRATION IN RAW WATER

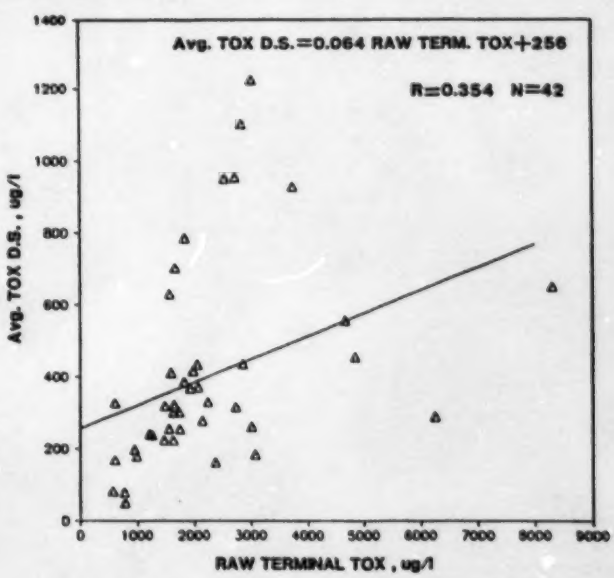


FIGURE 8. RELATIONSHIP BETWEEN INSTANTANEOUS TOTAL ORGANIC HALIDE CONCENTRATION IN DISTRIBUTION SYSTEM AND TERMINAL TOTAL ORGANIC HALIDE CONCENTRATION IN RAW WATER

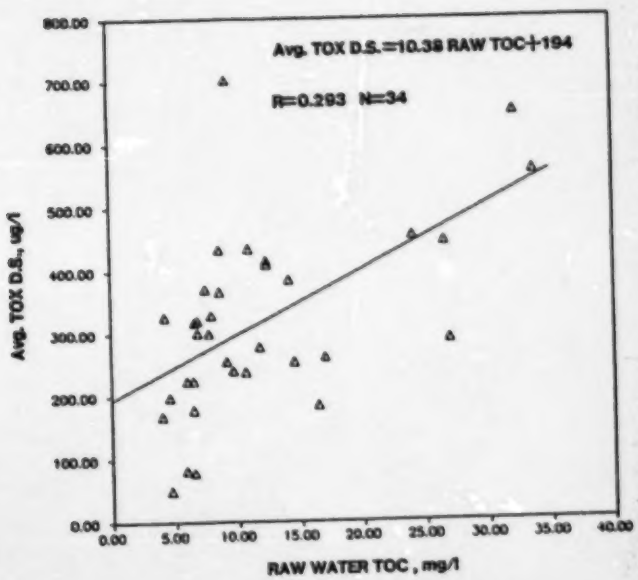


FIGURE 9. RELATIONSHIP BETWEEN INSTANTANEOUS TOTAL ORGANIC HALIDE CONCENTRATION IN DISTRIBUTION SYSTEM AND TOTAL ORGANIC CARBON CONCENTRATION IN RAW WATER FOR WATERS TREATED BY PRECIPITATIVE SOFTENING